

STATUS REPORT

**LOW-CONCENTRATION SURFACTANT FLOODING: PARTITIONING OF  
SYNTHETIC SURFACTANTS**

Project BE4A, Milestone 4, FY92 Annual Research Plan

By

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# **LOW-CONCENTRATION SURFACTANT FLOODING: PARTITIONING OF SYNTHETIC SURFACTANTS**

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## **ABSTRACT**

In this study, no demonstrable effect of alkaline additives on partitioning of synthetic surfactants between aqueous and oil phases was indicated. The partitioning of synthetic surfactants was distinctly different from the partitioning of carboxylic acid surfactants, which are pH dependent. Alkaline additives significantly reduced surfactant adsorption. Interfacial tension (IFT) levels very favorable to the mobilization of residual oil were achieved with dilute surfactant solutions that contained alkaline additives. With alkaline additives, it was easier to formulate very dilute (0.1 - 0.5%) surfactant solutions that have the properties to mobilize residual oil and minimize surfactant adsorption.

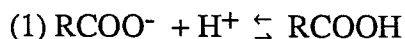
## **INTRODUCTION**

The objective of task 4 of the FY92 annual plan was to gain a better understanding of the surfactant partitioning that occurs when dilute surfactant mixtures (0.1-0.5%) and crude oils are contacted. One goal was to determine if, for optimized surfactant systems, there is a correlation between surfactant partitioning, IFT, surfactant adsorption, and the presence of weak alkaline additives. The partitioning of surfactants between the oil phase and aqueous phase was measured for several oils and chemical systems. This status report represents work performed for Project BE4A during the period October 1991 to June 1992.

The solvent properties of many surfactant systems are due predominantly to phenomena that result from formation of surfactant micelles. This characteristic of certain surfactant solutions is the primary contributor to the solubilization of oil that occurs when micellar solutions are used for enhanced oil recovery (EOR). Oil solubilization occurs when conditions allow aggregation of surfactant molecules into micelles. Above the critical micelle concentration (CMC), oil solubilization increases as surfactant concentration is increased. In micellar systems designed for oil recovery, most of the surfactant moves to a type III (as defined by Winsor) middle phase when oil is present and salinity is optimal. Below optimal salinity, most of the surfactant exists in the aqueous phase; above optimal, the surfactant and oil form a swollen upper oil phase.<sup>1-4</sup>

When surfactant concentration is low, the typical type III microemulsion phase is not formed, but significant oil recovery can still be achieved.<sup>5</sup> To increase the amount of oil mobilized (because true oil solubilization does not occur under these conditions), alkaline additives can be added to formulations that contain small amounts of surfactants. This process has been described as "cosurfactant-enhanced alkaline flooding."<sup>6</sup> The alkaline additives can cause changes in optimal salinity, reduce surfactant losses by adsorption, and have a beneficial effect on interfacial activity.<sup>7-11</sup> The beneficial effect of alkaline additives on IFT is especially pronounced when the crude oil contains acidic components.<sup>12</sup>

In addition to the effect that salt (ionic strength) has on surfactant partitioning, pH can also have a large effect. For oils that contain carboxylic acids, dissociation is strongly pH dependent. The equilibrium can be described by the equilibrium reaction:



However, the effect on phase transition has been shown to be much more complex than can be predicted from this simple equilibrium reaction. A good account of the complexity of the effect on phase transition was presented by Miller and Nelson.<sup>1,6,13</sup> Since the effect of pH has been shown to greatly complicate the understanding of chemical systems where carboxylic acids are present, acidic oils and fatty acid surfactants were purposely avoided for this work. This study was limited to synthetic surfactants (predominantly sulfonates) not derived from fatty acids. This greatly simplified interpretation of results.

Even though this work was performed mostly with non-acidic oils in order to eliminate effects due to acidic oil components, results were compared with those of previous work with acidic oil. In all cases, the concentration levels of the surfactants were not high enough to form type III microemulsions with the oils, although low IFT values were measured. Surfactant concentrations were kept low in order to narrow the area of study and to limit the study to surfactant systems that may be cost-effective at current crude oil prices.

When oil, water, and surfactant are in contact, surfactant molecules will be found in the oil phase, the aqueous phase, or in both phases. Surfactant partitioning between oil and aqueous phases is important to the economics of surfactant flooding because it will affect the amounts of surfactant that can be easily recovered from produced fluids and may affect surfactant losses due to adsorption onto reservoir rock. The total amount of surfactant adsorbed onto reservoir rock is at least partially related to the amount of surfactant that is injected into an oil reservoir. In this work, total surfactant loss during corefloods decreased as the amount of surfactant in the injected aqueous solution was decreased. Previous work has shown that the addition of alkaline additives

to surfactant solutions has a beneficial effect on IFT and reduces the amount of surfactant lost by adsorption on reservoir rock.<sup>5,7,12</sup> The primary objective of this work (task 4 of the annual plan) was to determine if the addition of alkaline additives to low-concentration surfactant mixtures affects the partitioning of surfactants between oil and water and to determine how this affects the economics of surfactant flooding.

## EXPERIMENTAL

### *Chemicals*

The chemical systems tested were composed of Stepan Petrostep B-110 surfactant (mol wt = 500), Petrostep B-105 surfactant (mol wt about 424), Chevron XP-100 surfactant (mol wt = 434), Shell Neodol 25-9 surfactant (mol wt = 610),  $\text{NaHCO}_3$ ,  $\text{Na}_2\text{CO}_3$ , and  $\text{NaCl}$ . All of the surfactants are anionic sulfonates, except Neodol 25-9 which is an ethoxylated alcohol. Some surfactant systems contained 2-butanol to reduce turbidity. One partitioning experiment was conducted with Wilmington (CA) oil, which is an acidic oil. The other experiments were conducted with a pure hydrocarbon, n-decane, and crude oil from Hepler (KS) oil field. The Tucker sand of Hepler field produces from a DOE Class I, fluvial dominated deltaic reservoir. The oil is, for practical purposes, non-acidic; the total acid number (TAN) is essentially zero.

### *Procedures*

Surfactant partitioning experiments were performed by contacting aqueous surfactant solutions with equal volumes of hydrocarbon. Partitioning between phases should be complete within 5 hours of mixing.<sup>14</sup> To ensure complete equilibration, samples were gently agitated once each day for about 2 weeks. A Pasteur pipette was used to separate the aqueous phase for surfactant analysis. Aqueous phases were analyzed for anionic surfactants with two-phase methylene blue titration, using CTAB as the titrant.<sup>15</sup> The nonionic surfactant concentration was determined by the colorimetric response of a cobalt thiocyanate-nonionic surfactant complex.<sup>16</sup>

Surfactant adsorption experiments were performed by contacting 5 g of crushed core with 10 mL of surfactant solution. After agitating the samples continuously for 3 weeks, a portion of the aqueous phase was removed and then centrifuged to remove fines. After centrifugation, the samples were analyzed for surfactant using the procedures described above.

Salinity scans were conducted by varying the concentration of  $\text{NaCl}$ ,  $\text{NaHCO}_3$ , or  $\text{Na}_2\text{CO}_3$ . The IFT of each oil/chemical system was measured with a University of Texas spinning drop interfacial tensiometer. This procedure was previously described.<sup>17</sup>

## DISCUSSION

Several series of experiments were performed to determine the effect of alkaline additives on surfactant partitioning, surfactant adsorption, and IFT. One series of experiments was conducted with a pure hydrocarbon, n-decane, and a surfactant mixture that contained 0.25% Petrostep B-110 surfactant and 0.15% Petrostep B-105 surfactant. Ionic strength adjustment (salinity scan) was accomplished with NaCl. The results obtained for NaCl were compared with the results obtained with NaHCO<sub>3</sub>, a very weak alkaline additive (pH 8.3). Experiments were also conducted with Na<sub>2</sub>CO<sub>3</sub>, a mildly alkaline additive (pH 10.8).

The results of NaCl, NaHCO<sub>3</sub>, and Na<sub>2</sub>CO<sub>3</sub> scans are shown, respectively, in Figs. 1, 2, and 3. When salinity scans were conducted with NaCl, NaHCO<sub>3</sub>, and Na<sub>2</sub>CO<sub>3</sub>, most of the surfactant was contained in the aqueous phase at low salinities. As salinity was increased, the surfactants partitioned into the decane. There was a marked decrease in initial IFT when the surfactant began to partition from the aqueous phase to the decane phase. Initial IFT is the IFT measured at short time (1 to 2 min.) during dynamic IFT measurement. Minimum IFT achieved during dynamic IFT measurement also showed a marked reduction when the surfactant partitioned into the decane. As salinity was changed, no pronounced minimums were observed; instead, IFT values remained low even at high salinities. Comparison of the three systems shows that the general trend was for IFT to decrease as surfactant began to partition into the decane phase and

$$\text{IFT}_{\text{Na}_2\text{CO}_3} < \text{IFT}_{\text{NaHCO}_3} < \text{IFT}_{\text{NaCl}}$$

For all three systems, adsorption increased as salinity was increased. Adsorption was lowest with sodium bicarbonate, about 40% less than that with either sodium chloride or sodium carbonate.

Salinity scans were used to optimize the surfactant system described above with crude oil from Hepler (KS) oil field. The major difference between the results obtained with crude oil and with decane was that, with crude oil, very little surfactant partitioned into the oil as salinity was increased. This was true for NaCl, NaHCO<sub>3</sub>, and Na<sub>2</sub>CO<sub>3</sub> scans. These results are shown, respectively, in Figs. 4, 5, and 6.

Low IFT values were measured for NaCl and Na<sub>2</sub>CO<sub>3</sub> scans conducted with Hepler oil and the 0.25% Petrostep B-110/0.15% Petrostep B-105 surfactant mixture. It was previously demonstrated that very good oil recoveries can be achieved with an equi-normal mixture of NaHCO<sub>3</sub> and Na<sub>2</sub>CO<sub>3</sub> and this surfactant mixture. Oil recoveries were less when only NaCl was

used to adjust the ionic strength of the surfactant formulation. As shown in Fig. 5, IFT values for  $\text{NaHCO}_3$  were unexpectedly high, and there is no obvious explanation for this result. The  $\text{NaHCO}_3$  formulation was not tested for oil recovery. The values shown in Figs. 4, 5, and 6 for surfactant adsorption are the same values for this surfactant system presented in Figs. 1, 2, and 3.

The most interesting result for the surfactant mixture that contained 0.25% Petrostep B-110 and 0.15% Petrostep B-105 is the difference between surfactant partitioning with n-decane and Hepler oil. It appears that this surfactant mixture is soluble in n-decane at high salinities and insoluble in Hepler oil at all salinities. Despite the insolubility in the crude oil, very low IFT values and good oil recovery were obtained with this surfactant system and Hepler crude oil.

Surfactant partitioning was also measured for a second surfactant mixture and Hepler oil. This mixture contained 0.5% Chevron XP-100 surfactant. Salinity scans were conducted with NaCl and  $\text{NaHCO}_3$ . Only two concentrations of NaCl were tested, 0.5 and 1.0% NaCl (Fig. 7). Most of the surfactant was partitioned into the aqueous phase at both salinities. Figure 8 shows the  $\text{NaHCO}_3$  scan. Most of the surfactant was partitioned into the aqueous phase up to 2.4%  $\text{NaHCO}_3$ , where the solubility of the surfactant in brine became so low that precipitation occurred. IFT went through a minimum at 1.2%  $\text{NaHCO}_3$ , but was very low at all salinities. Surfactant adsorption was exceedingly low. Adsorption also showed a slight minimum at 1.2%  $\text{NaHCO}_3$ .

All of the experiments described above were performed with non-acidic oils and anionic surfactants. One experiment was also performed with an acidic oil and a nonionic surfactant, Shell Neodol 25-9 ethoxylated alcohol. The oil was from Wilmington (CA) oil field. This surfactant and oil were previously optimized for lowest IFT using an equi-normal mixture of  $\text{NaHCO}_3$  and  $\text{Na}_2\text{CO}_3$  for the salinity scan.<sup>12</sup> It is probable that some naturally occurring carboxylic acid surfactants from the crude oil are present in the aqueous phase, especially at elevated pH. However, the natural surfactants are present at such low levels (compared to the synthetic surfactant) that no information was gained about the partitioning of the natural surfactants. The results of a partition measurement at optimum conditions are shown in Fig. 9. In this case, almost all of the synthetic surfactant was shown to be in the aqueous phase. This was the same type of surfactant partitioning that was seen with the other crude oil and surfactant systems described above.

## SUMMARY AND CONCLUSIONS

It appears that the economics of surfactant flooding with low surfactant concentrations are not greatly affected by partitioning of synthetic surfactant between oil and aqueous phases. In

this study, no demonstrable effect of alkaline additives on the partitioning of synthetic surfactant between aqueous and oil phases was observed. The results do show (1) that alkaline additives significantly reduce surfactant adsorption and (2) that IFT levels very favorable to the mobilization of residual oil can be achieved with dilute surfactant solutions that contain alkaline additives. With alkaline additives, very dilute (0.1 to 0.5%) surfactant solutions that have the properties to mobilize residual oil and minimize surfactant adsorption can be easily formulated.

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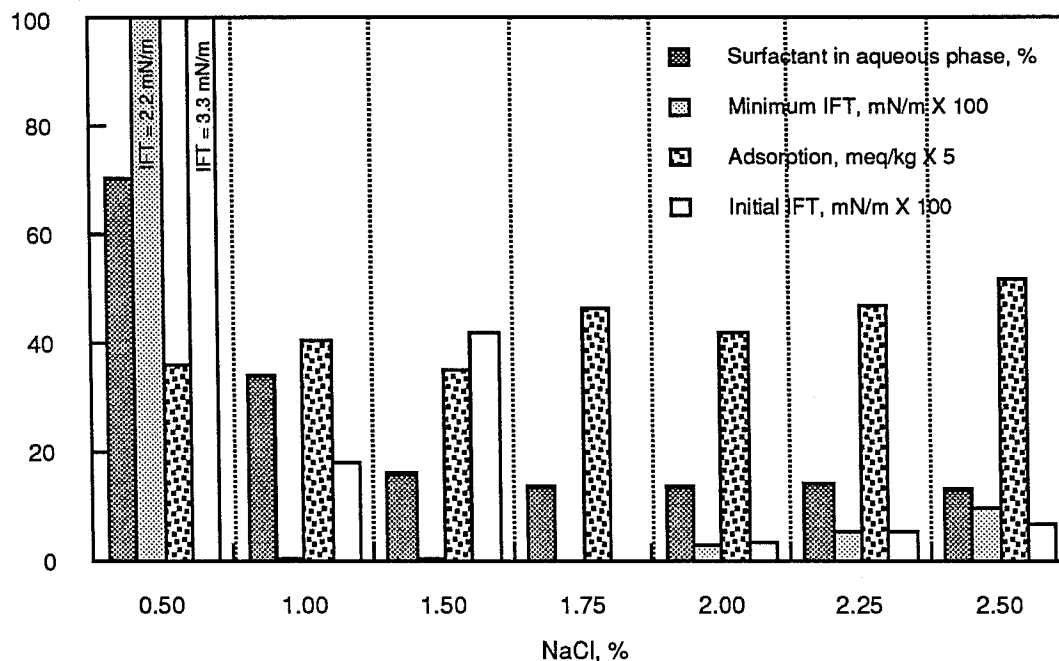


FIGURE 1. - IFT, adsorption, and surfactant partitioning for n-decane and a mixture of 0.25% Petrostep B-110 + 0.15% Petrostep B-105 surfactants and NaCl, 23° C.

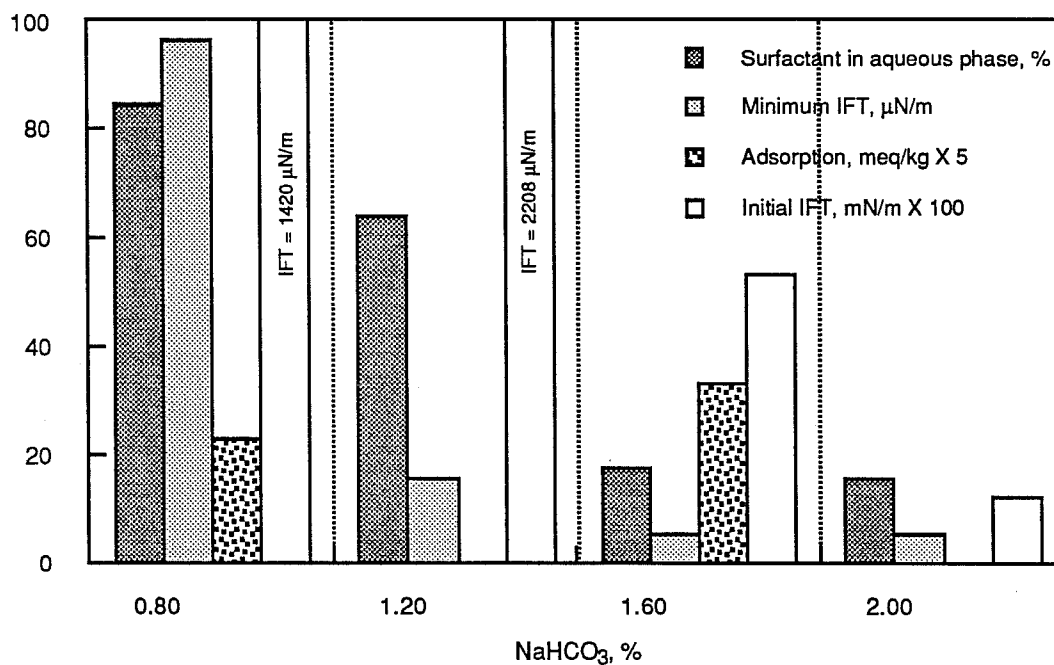


FIGURE 2. - IFT, adsorption, and surfactant partitioning for n-decane and a mixture of 0.25% Petrostep B-110 + 0.15% Petrostep B-105 surfactants and NaHCO<sub>3</sub>, 23° C.

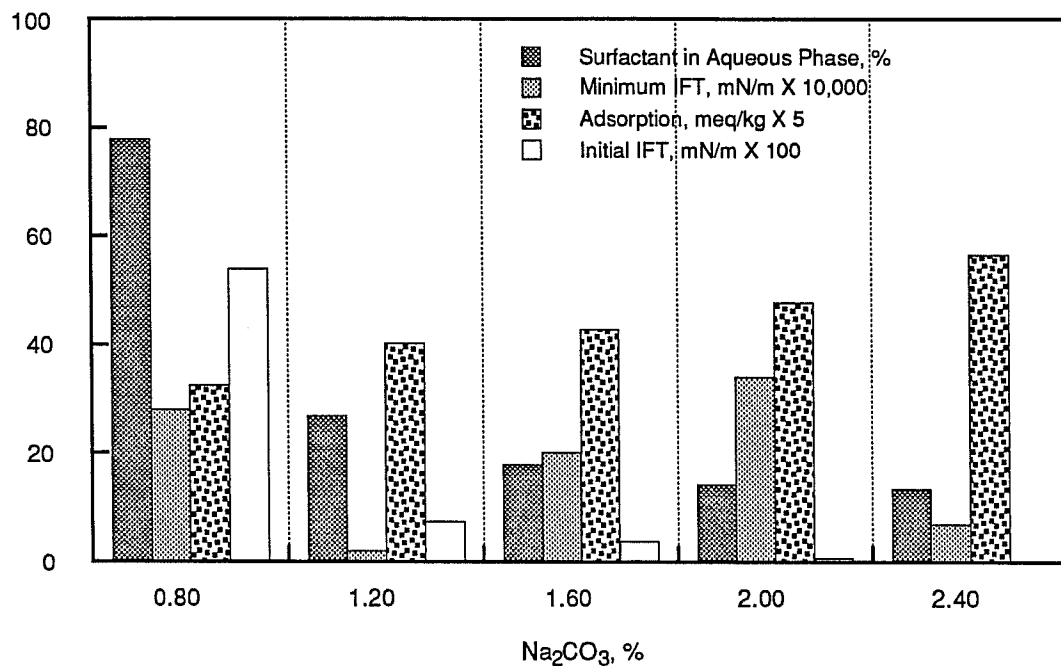


FIGURE 3. - IFT, adsorption, and surfactant partitioning for n-decane and a mixture of 0.25% Petrostep B-110 + 0.15% Petrostep B-105 surfactants and Na<sub>2</sub>CO<sub>3</sub>, 23° C.

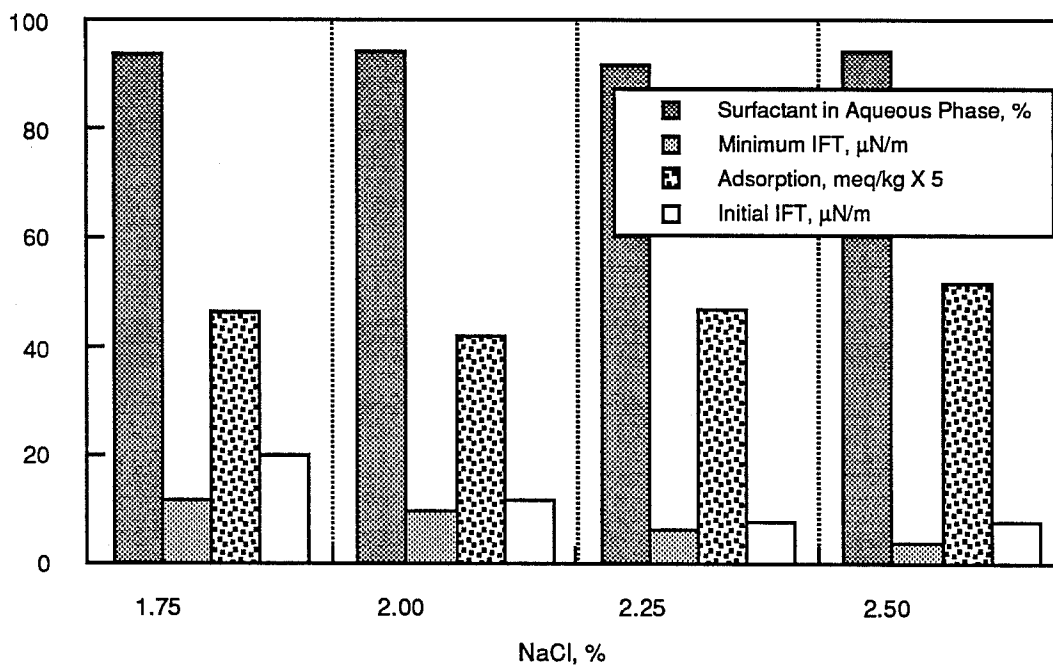


FIGURE 4. - IFT, adsorption, and surfactant partitioning for Hepler oil and a mixture of 0.25% Petrostep B-110 + 0.15% Petrostep B-105 surfactants and NaCl, 23° C.

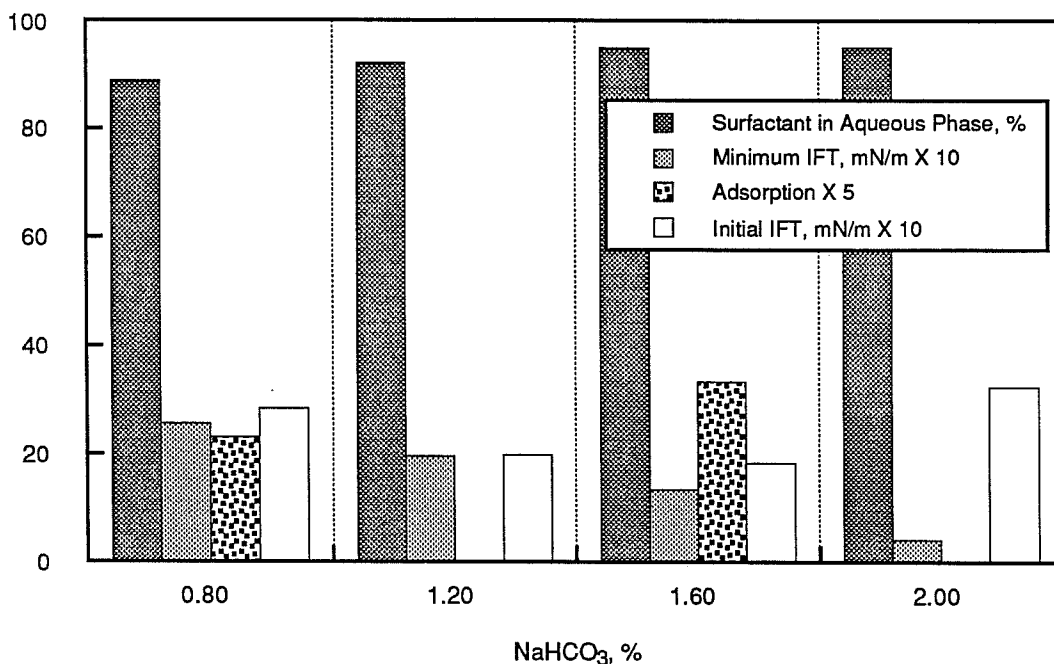


FIGURE 5. - IFT, adsorption, and surfactant partitioning for Hepler oil and a mixture of 0.25% Petrostep B-110 + 0.15% Petrostep B-105 surfactants and NaHCO<sub>3</sub> 23° C.

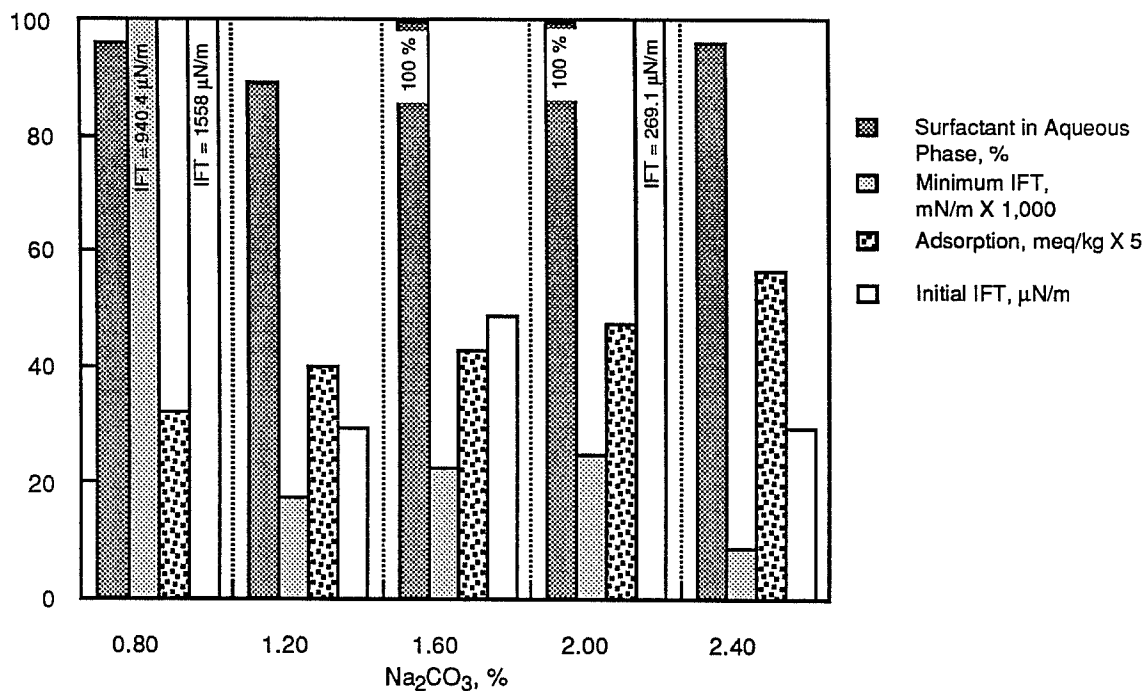


FIGURE 6. - IFT, adsorption, and surfactant partitioning for Hepler oil and a mixture of 0.25% Petrostep B-110 + 0.15% Petrostep B-105 surfactants and Na<sub>2</sub>CO<sub>3</sub> 23° C.

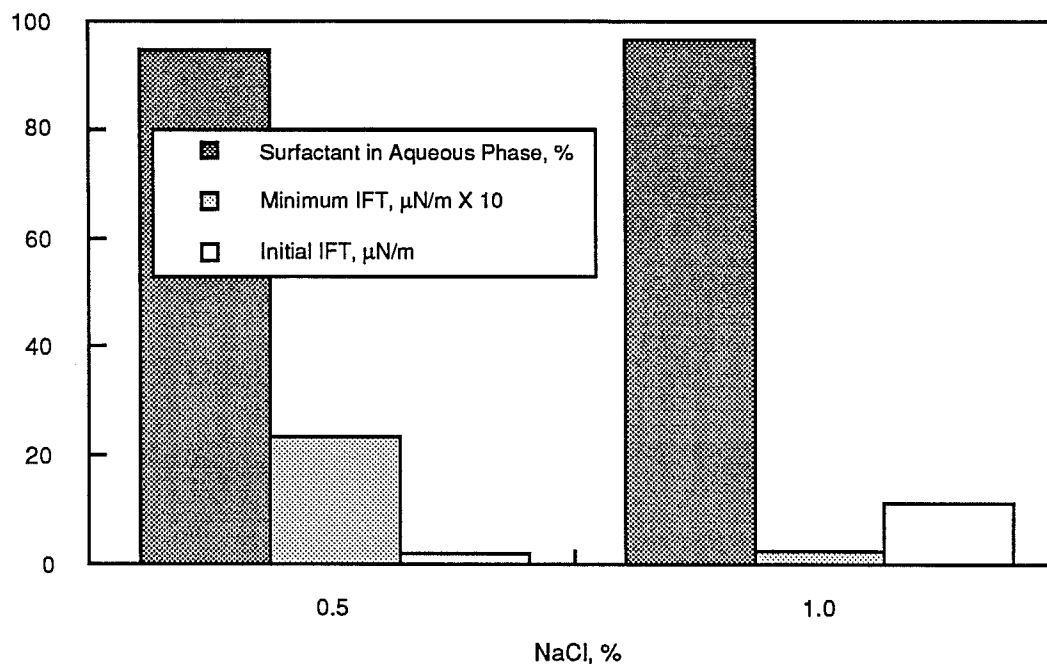


FIGURE 7. - IFT and surfactant partitioning for Hepler oil and a mixture of 0.5% Chevron XP-100 surfactant, 0.45% sodium tripolyphosphate (STPP), and NaCl, 23° C.

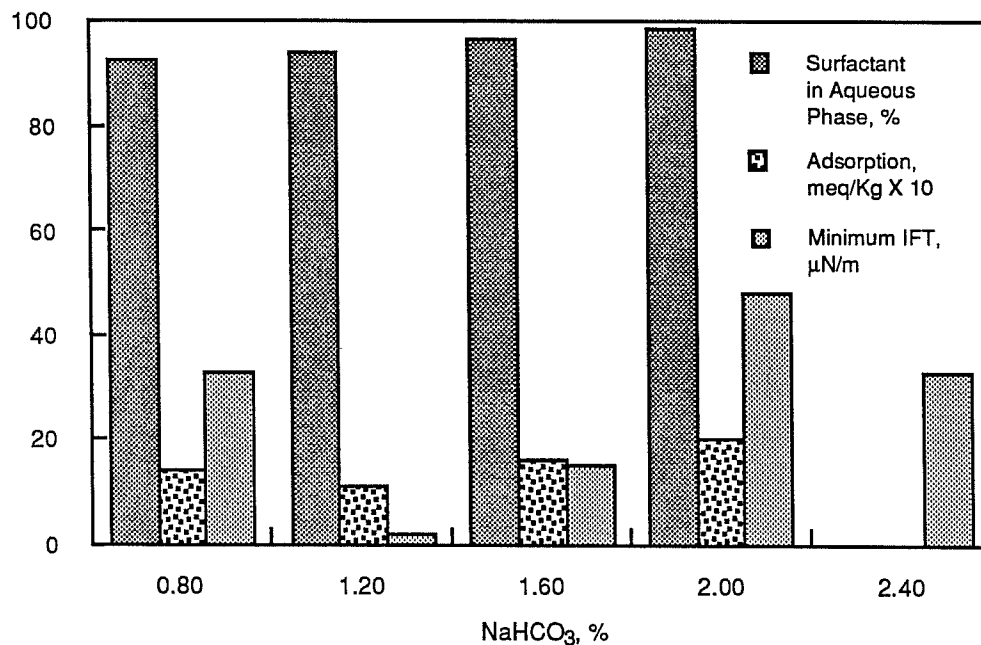


FIGURE 8. - IFT, adsorption, and surfactant partitioning for Hepler oil and a mixture of 0.5% Chevron XP-100 surfactant, 0.45% sodium tripolyphosphate (STPP), and  $\text{NaHCO}_3$ , 23° C.

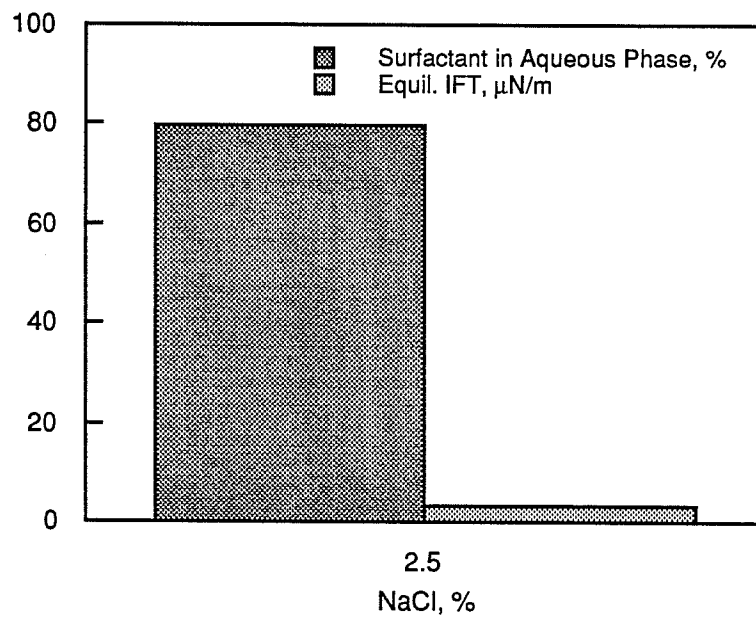


FIGURE 9. - IFT and surfactant partitioning for Wilmington oil and a mixture of 0.1% Neodol 25-9 surfactant, 0.8%  $\text{NaHCO}_3$ , 0.5%  $\text{Na}_2\text{CO}_3$  (pH 9.5 carbonate mixture) and 2.5% NaCl, 23° C.

## APPENDIX A

### Data Presented in Figures 1-9

		Surfactant in aqueous phase, %	Initial IFT, $\mu\text{N/m}$	Minimum IFT, $\mu\text{N/m}$	Adsorption, meq/kg
Figure 1	<u>NaCl, %</u>				
	0.50	70.4	3337.2	2202.9	7.21
	1.00	34.2	181.8	5.4	8.12
	1.50	16.3	421.3	7.1	7.09
	1.75	13.7	--	--	9.30
	2.00	13.6	35.2	29.3	8.44
	2.25	14.2	54.2	54.2	9.38
	2.50	13.4	68.7	99.8	10.37
Figure 2	<u>NaHCO<sub>3</sub>, %</u>				
	0.50	84.7	1419.8	96.6	4.63
	1.00	64.2	2207.9	15.9	--
	1.50	17.9	531.9	5.7	6.69
	1.75	15.8	124.9	5.5	--
Figure 3	<u>Na<sub>2</sub>CO<sub>3</sub>, %</u>				
	0.80	77.8	540.8	2.8	6.49
	1.20	26.9	72.7	0.2	8.04
	1.60	17.8	37.3	2.0	8.55
	2.00	14.2	7.7	3.4	9.54
	2.40	13.3	0.7	0.7	11.33
Figure 4	<u>NaCl, %</u>				
	1.75	94.0	20.0	12.0	9.30
	2.00	94.5	12.0	10.0	8.44
	2.25	91.9	8.0	6.5	9.38
	2.50	94.3	8.0	4.0	10.37
Figure 5	<u>NaHCO<sub>3</sub>, %</u>				
	0.80	88.8	2848.7	2562.6	4.63
	1.20	92.1	1990.6	1974.3	--
	1.60	95.0	1845.8	1336.6	6.69
	2.00	95.1	3247.5	411.0	--

# APPENDIX A (Continued)

## Data Presented in Figures 1-9

		Surfactant in aqueous phase, %	Initial IFT, $\mu\text{N/m}$	Minimum IFT, $\mu\text{N/m}$	Adsorption, meq/kg
Figure 6	<u><math>\text{Na}_2\text{CO}_3</math>, %</u>				
	0.80	96.3	1558.0	940.4	6.49
	1.20	89.5	29.5	17.8	8.04
	1.60	100.0	48.7	22.8	78.55
	2.00	100.0	269.1	25.0	9.54
	2.40	96.2	29.7	9.0	11.33
Figure 7	<u><math>\text{NaCl}</math>, %</u>				
	0.50	94.9	2.4	2.4	--
	1.00	96.7	11.5	0.3	--
Figure 8	<u><math>\text{NaHCO}_3</math>, %</u>				
	0.80	92.9	34.0	33.0	1.40
	1.20	94.0	66.5	2.0	1.10
	1.60	97.0	20.3	15.0	1.60
	2.00	98.8	49.4	48.0	2.00
	2.40	--	230.4	33.0	--
Figure 9	<u><math>\text{NaCl}</math>, %</u>				
	2.50	80.1	--	3.3	--